1,051,735

PATENT SPECIFICATION

DRAWINGS ATTACHED

1,051

Inventors: JOHN WALFORD McLEAN and THOMAS HENRY HUGHES

Date of filing Complete Specification: May 6, 1964.
Application Date: May 8, 1963. *No. 18192/63.*
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Index at acceptance:—C1 J (2, 9, 10, 12, 14, 19, 33); A5 R75B
Int. Cl.:—C 04 b 35/00 // A 61 c

COMPLETE SPECIFICATION

Improvements in Dental Materials

We, NATIONAL RESEARCH DEVELOPMENT CORPORATION, a Company organised under the laws of Great Britain, of 1, Tilney Street,

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ERRATA

SPECIFICATION No. 1,051,735

Page 2, line 53, *for* "Serial No. 1004376" *read* "Serial No. 1,105,111"
Page 4, line 3, *for* "Serial No. 1004376" *read* "Serial No. 1,105,111."

THE PATENT OFFICE
5th February 1968

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Artificial teeth are normally manufactured from porcelain having a high fusing temperature, e.g. 1300° C, and platinum or gold clad pins are normally fired or welded into the back of the teeth to form attachments to the denture base.

Porcelain jacket crowns are constructed on platinum matrices using suitably pigmented porcelain powder which is generally bonded with distilled water to facilitate moulding. The platinum matrix with the hand moulded jacket crown is fired in a furnace, ground to shape and then refired to produce a glaze similar to human dental enamel. Dental inlays are produced in a similar manner using a platinum matrix or a refractory mould.

Dental porcelain has the disadvantage of mechanical strength limitations. The strength of this porcelain, which is generally measured in terms of the modulus of rupture, is very low e.g. 4000—12000 pounds per square inch. This lack of mechanical strength makes the material unsuitable for use in areas of of the mouth where high stress concentrations are prevalent. The brittle nature of dental porcelain currently used is also manifest by the high breakage rate of artificial teeth on

According to the invention, a method constructing denture work and dental restoi tions comprises moulding into the form a tooth or an integral part of a tooth refractory oxide composition comprising least 60% by weight of alumina, titania zirconia, or a mixture thereof, the balan except for impurities and incidental cons tuents being silica, and firing it at a ter perature in the range 1300—1750° C to bri: about sintering and recrystallization of t oxide without substantial fusion thereof.

By 'impurities and incidental constituent are meant those impurities and constituer that may occur in minor quantities in cor mercially available refractory oxides, such other refractory oxides, iron oxides, and flu ing agents such as alkali metal and alkali earth silicates.

The invention also extends to artifici teeth, reinforcements therefor, denture plat jacket crowns, dental inlays and other dentu work made by the method of the invention.

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COMPLETE SPECIFICATION

Improvements in Dental Materials

We, NATIONAL RESEARCH DEVELOPMENT CORPORATION, a Company organised under the laws of Great Britain, of 1, Tilney Street, London, W.1, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to methods and materials for use in the construction of denture work and dental restorations, e.g. artificial teeth and reinforcements therefor, jacket crowns and inlays.

The material commonly used for this purpose is known as dental porcelain, and is composed of a mixture of feldspar, silica and kaolin in various proportions together with a small amount of a fluxing material.

Artificial teeth are normally manufactured from porcelain having a high fusing temperature, e.g. 1300° C, and platinum or gold clad pins are normally fired or welded into the back of the teeth to form attachments to the denture base.

Porcelain jacket crowns are constructed on platinum matrices using suitably pigmented porcelain powder which is generally bonded with distilled water to facilitate moulding. The platinum matrix with the hand moulded jacket crown is fired in a furnace, ground to shape and then refired to produce a glaze similar to human dental enamel. Dental inlays are produced in a similar manner using a platinum matrix or a refractory mould.

Dental porcelain has the disadvantage of mechanical strength limitations. The strength of this porcelain, which is generally measured in terms of the modulus of rupture, is very low e.g. 4000—12000 pounds per square inch. This lack of mechanical strength makes the material unsuitable for use in areas of of the mouth where high stress concentrations are prevalent. The brittle nature of dental porcelain currently used is also manifest by the high breakage rate of artificial teeth on denture plates when they are accidentally dropped on to hard surfaces and by the fractures which occurs in dental crowns subjected to heavy biting forces.

Attempts have been made to improve the strength of dental porcelain by firing water bonded powder in a vacuum thus eliminating entrapped air bubbles in the fired porcelain. Other techniques to reinforce dental porcelain involve the use of a platinum-palladium-gold alloy with a melting range of 1150—1175° C to construct a reinforced core or backing onto which a dental porcelain of a similar coefficient of thermal expansion is fired. This latter technique has been used to construct dental crowns and bridgework permanently cemented to existing teeth in the mouth.

According to the invention, a method of constructing denture work and dental restorations comprises moulding into the form of a tooth or an integral part of a tooth a refractory oxide composition comprising at least 60% by weight of alumina, titania or zirconia, or a mixture thereof, the balance except for impurities and incidental constituents being silica, and firing it at a temperature in the range 1300—1750° C to bring about sintering and recrystallization of the oxide without substantial fusion thereof.

By 'impurities and incidental constituents' are meant those impurities and constituents that may occur in minor quantities in commercially available refractory oxides, such as other refractory oxides, iron oxides, and fluxing agents such as alkali metal and alkaline earth silicates.

The invention also extends to artificial teeth, reinforcements therefor, denture plates, jacket crowns, dental inlays and other denture work made by the method of the invention.

Although the exact nature of the sintering and recrystallization process is not entirely certain, it appears that during firing the following steps occur. First, a welding effect occurs at the points of contact between adja-

cent oxide particles, giving rise to a lensing effect, as normally occurs in sintering processes. Migration of atoms then takes place from one particle to the other, resulting in a shift in the particle boundaries, or "recrystallization." During recrystallization, the shift in grain boundaries results in the formation of a closely interlocking crystalline structure of considerable strength, the improved packing of the particles resulting in shrinkage of the oxide mass. The resulting recrystallized oxide has mechanical properties much superior to those of dental porcelain which are limited by the inferior mechanical characteristics of the glass phase.

Although alumina is the preferred refractory oxide, titanium dioxide, zirconia and mixed oxides of alumina and silica are also particularly useful.

The firing temperature required varies according to the oxide used, and to some extent according to the time for which the oxide is fired. In the case of substantially pure titanium dioxide, the firing temperature would be in the region of 1400° C. Pure alumina requires a firing temperature of 1750° C, but by slightly reducing the purity of the alumina the temperature necessary for recrystallization can be lowered. Such alumina of a lower degree of purity may advantageously be used with consequent reduction in the necessary firing temperature: for example by using alumina of about 85% purity, the balance being principally silica, recrystallization takes place at about 1500° C, whilst a mixture comprising 60% alumina, the balance being mainly silica, may be fired at 1300° C. This enables cheaper and simpler furnaces to be used for the firing process.

The oxide after firing has a smooth surface impermeable to oral fluids. This surface may be further glazed and pigmented by any known means.

Refractory oxides may be utilized according to the method of the invention in the following applications:

1. As a mouldable material which is subsequently fired to make dental jacket crowns.

2. To make preformed recrystallized oxide reinforcements for dental porcelain, or aluminous porcelain as described in our copending application No. 39709/63, Serial No. 1,004,376, e.g.

a) As a backing material onto which dental porcelain may be fired to form dental jacket crowns,

b) As a preformed bridge pontic onto which a porcelain facing may be fired,

c) As a reinforcing core for artificial porcelain teeth.

3. As a mouldable material to be fired to manufacture complete dental bridgework for cementing on to natural human teeth.

4. As a mouldable material for application when fired in the manufacture of artificial teeth, molars, pre-molars and incisors.

5. As a mouldable material to be fired into preformed shapes (dental inlays) for cementing into individual human teeth.

6. As a facing material to be fired onto metal posts to carry backings of e.g. molybdenum.

It will be readily appreciated that the cost of the recrystallized oxide as a reinforcing material will be considerably less than that of the precious metals currently in use for this purpose.

The following examples illustrate the invention. Although the examples relate to the use of alumina, it should be understood that other oxides could be substituted with the appropriate adjustment of firing temperatures.

In the examples, the alumina was moulded by any known means into the required shape before being fired. In all the examples a binder was used to give cohesion to the alumina mass during the moulding process. The binder should normally be such as to leave no residue after firing, and suitable binders of this character are well known to those skilled in the art; methyl cellulose was used in the following example.

EXAMPLE 1.

A mixture of 10% by weight of methyl cellulose and the balance water was made as follows. The methyl cellulose was mixed with half the required amount of water and heated to 80° C for five to ten minutes with continuous stirring until it was wetted. The mixture was cooled to 20° C when the remainder of the water was gradually added until dispersion of the methyl cellulose was complete.

The alumina and prepared methyl cellulose gel may then be mixed in various proportions according to the nature of the alumina to obtain a mouldable mass; in this example 200 grams alumina were mixed with 30 grams of methyl cellulose gel. To facilitate release from the mould and to improve the moulding characteristics a suitable releasing agent, which agents are well known in the art, may be added to the mixture. A suitable proportion is 30 ml of agent to 200 grams alumina. The mixture was then moulded to the required shape. After release from the mould, the moulded alumina was first dried slowly in an oven at 200° C and then heated to the required firing temperature.

The procedure was conducted twice, firstly with alumina of 95% purity and secondly with alumina of 85% purity.

Articles moulded from alumina of 95% purity, the impurities being mainly silica with minor quantities of titania, iron oxide and fluxing agents such as alkali metal or alkaline earth silicates, were heated for two hours at 1650° C. Comparative properties of the material thus obtained and of dental porcelain are illustrated in the following table.

Physical Properties	Recrystallized alumina	Porcelain
Tensile strength lb/sq. in.	17,200	5,000
Compressive strength lb/sq. in.	316,000	50,000
Young's Modulus lb/sq. in.	46.1×10^6	
Modulus of Rupture lb/sq. in.	55,000	3700—12,000
Coefficient of Expansion		
0 to 200° C. $\times 10^{-6}$/°C.	6.2	6.4 to 7.8
0 to 400° C. "	6.7	
0 to 600° C. "	7.1	
0 to 800° C. "	7.5	
0 to 1,000° C. "	7.9	
Thermal Conductivity, 100° C. joule cm/cm²/sec./°C.	0.162	0.010
Water absorption	nil	0 to 2%
Hardness Moh's scale	9	
Rockwell C scale	72	42
Vickers scale at 10 kg load VPN	1,200	430

The coefficient of expansion of human teeth is in the order of 11.4×10^{-6}/°C.

Articles moulded from alumina of 85% purity, the impurities being mainly free silica with such minor quantities of titania, iron oxide and fluxing agents such as alkali metal or alkaline earth silicates as normally occur in commercially available oxides, were fired for two hours at 1500° C and were found to be of only slightly inferior physical properties to the recrystallized alumina in the above table.

The procedure was repeated using a mixed oxide consisting of approximately 60% alumina and about 40% silica with minor proportions of impurities such as titania, iron oxide, and fluxing agents. The mixed oxide was heated for 2 hours at 1300° C.

Examples of teeth for denture work and dental restorations made by the method of the invention are described below, with reference where appropriate to the accompanying drawings in which:

Fig. 1 is a vertical cross section of a tooth mould containing an artificial posterior tooth incorporating a preformed recrystallized alumina core made by the method of the invention.

Fig. 2 is a vertical cross section of another tooth mould containing an artificial anterior tooth also incorporating a preformed alumina core.

Fig. 3 shows in vertical cross-section a post crown incorporating an alumina tube fitted to the root of a human tooth.

Fig. 4 shows in vertical cross-section a jacket crown similarly fitted.

Fig. 5 is a horizontal cross-section of an anterior veneer facing for bridgework.

EXAMPLE 2.

Recrystallized alumina may be nickel plated by the steps of impregnating the surface of a recrystallized alumina component with molybdenum paint, firing the paint on to the surface of the alumina at a temperature exceeding 1600° C, nickel plating the resultant surface using standard plating methods, and then soldering gold backings onto the nickel plating, again by conventional methods.

EXAMPLE 3.

Alumina cores manufactured by the method of the invention are built up to the required

tooth form with a layer of the aluminous porcelain described in our copending application No. 39709/63, Serial No. 1,004,376, or with dental porcelain having substantially the same coefficient of expansion, and are glazed with a veneer of feldspathic enamel. The method of making posterior and anterior teeth using this principle is illustrated in figure 1 and 2 respectively. A metal mould made in two sections 1 and 2 is constructed with a metal spigot 3 projecting into the mould cavity. A preformed recrystallized alumina core 4 of 1.5 to 2.5 millimetres thickness depending on the size of the tooth is slipped onto the spigot 3. The core should fit the spigot with a tolerance of about 0.01 inches. A veneer of feldspathic enamel is first applied to the inner surface of he mould cavity. Dental porcelain or aluminous porcelain containing a suitable pigment, and admixed if necessary with an inorganic binder, is then charged into the mould to form a layer 5 preferably of thickness 0.7 to 1.0 millimetres around the alumina core. The two halves of the mould are then closed in a hot press and the complete tooth fired and completed by standard dental manufacturing methods which include the application of an enamel veneer 6.

The procedures described in this example are particularly suitable for use when it is desired to use pigments which may not be stable at temperatures of 1500° C or above, and therefore cannot be directly incorporated in recrystallized alumina components. Teeth made by these procedures may be used both for bridge pontics and on artificial dentures.

EXAMPLE 4

Recrystallized alumina may also be used for the construction of post crowns as illustrated in figure 3. A 1 to 1.5 millimetres diameter stainless steel post 10 is fitted to the root canal 11 of an existing tooth, and recrystallized alumina tube 12 is ground to fit over the exposed portion 13 of the post. The post 10 and tube 12 may be made in various sizes but the tube should fit the post accurately. An impression is taken by any standard dental procedure of the root surface 14 with only the post in position. A copper or silver die is made up from the impression and a platinum matrix is applied to the root surface 14. A dental crown is constructed by firing an aluminous porcelain body 15 onto the alumina tube 12 in the die. Feldspathic dental enamel 16 is applied at a second firing. The alumina tube 12 gives reinforcement to the porcelain 15 and eliminates the necessity of casting a gold core for the crown.

EXAMPLE 5.

A reinforced porcelain jacket crown containing a preformed recrystallized alumina backing may be made by manufacturing a copper or silver model stump of the jacket crown and forming a platinum matrix using the model stump. A thin coat of pigmented aluminous porcelain is then applied to the palatal aspect of the platinum matrix and a preformed alumina backing vibrated into position. The body colour is built up onto the remainder of the matrix with aluminous porcelain and the whole is then fired to produce a jacket crown. Feldspar enamel is then applied to the appropriate surfaces of the crown and the whole refired: the resultant crown is shown in Figure 4 in position on an existing human tooth 20, the jacket crown comprising the pigmented aluminous porcelain layer 21 which lay against the palatal aspect. of the platinum matrix, the preformed alumina backing 22, the pigmented aluminous porcelain 23 and the feldspar enamel 24. The crown described in this example has the advantage of strength on the palatal surface 25 which is the most common site for fracture during mastication.

EXAMPLE 6.

As a result of the refractory nature of recrystallized alumina it has been found possible to cast dental gold directly on to this material. Bridge pontics are constructed using recrystallized alumina cores covered with aluminous porcelain having a firing temperature in excess of 1100° C. Wax is applied to the palatal retention areas of the preformed alumina core and a gold casting is made by standard dental procedure using the wax impressions. This technique may be used to construct one piece bridgework in which the abutment inlays and alumina pontic may be joined with a gold casting made by the wax process.

EXAMPLE 7.

An interior veneer facing for bridgework may be made up as illustrated in figure 5. An alumina backing 30 is provided with a dovetail 31 by which it may be attached to the bridgework and a layer 33 of suitably pigmented aluminous porcelain fired to its anterior surface 32, after which a veneer 34 is applied over the aluminous porcelain.

EXAMPLE 8.

Preformed strips of recrystallized alumina 0.6 millimetres thick were prepared to fit the palatal aspect of a copper plated model stump prepared by recognised procedures for manufacturing dental jacket crowns. Dental porcelain (firing temperature 980° C) was then fired onto the recrystallized alumina backing and a jacket crown completed by the normal technique used in a dental laboratory.

In this manner a desirable natural tooth like appearance was achieved using the alumina

backing as a reinforcing material. The result was vastly superior in appearance to opacifying effect of the platinum palladium alloys which are normally used for reinforcing purposes.

Although the above examples use alumina as the refractory oxide, it will of course be appreciated that the alumina can be replaced with other refractory oxides such as titania, zirconia and mixed oxides of alumina and silica.

WHAT WE CLAIM IS:—

1. A method of constructing denture work and dental restorations comprising moulding into the form of a tooth or an integral part of a tooth a refractory oxide composition comprising at least 60% by weight of alumina, titania or zirconia, or a mixture thereof, the balance except for impurities and incidental constituents being silica, and firing it at a temperature in the range of 1300—1750° C to bring about sintering and recrystallization of the oxide without substantial fusion thereof.

2. A method according to claim 1 in which the refractory oxide composition contains minor quantities of fluxing agents.

3. A method according to claim 1 or 2 in which the composition contains 85% or more of alumina.

4. A method according to claim 3 in which the firing temperature is between 1500° and 1750° C.

5. A method according to any of the preceding claims in which a binder is added to the unfired oxide to facilitate moulding.

6. A method according to any of the preceding claims in which a releasing agent is added to the unfired oxide prior to moulding to facilitate release from the mould.

7. A method according to any of the preceding claims in which a layer of dental porcelain is subsequently fired onto the surface of the fired recrystallized oxide component.

8. A method according to any of claims 1—6 in which a layer of aluminous porcelain is subsequently fired onto the surface of the fired recrystallized oxide component.

9. A method as claimed in claim 7 or 8 in which a layer of dental enamel or veneer is fired onto the surface of the porcelain.

10. Denture work or dental restorations made by the method of any of claims 1 to 9.

11. An artificial tooth comprising a core of a sintered and recrystallized refractory oxide composition made according to any of claims 1—6, a layer of dental or aluminous porcelain fired onto the core, and a veneer covering at least part of the porcelain layer.

12. A post crown comprising a tube of sintered and recrystallized refractory oxide composition made according to any of claims 1—6, so shaped as to fit a metal post fitted to the root canal of an existing human tooth, a layer of dental or aluminous porcelain covering the exposed surfaces of the tube, and a veneer covering at least part of the surface of the porcelain layer.

13. A jacket crown comprising aluminous porcelain, the palatal face of which contains a preformed backing of a sintered and recrystallized refractory oxide composition made according to any of claims 1—6, onto which the porcelain is fired in a platinum matrix prepared from a copper or silver model stump of the jacket crown.

14. Artificial teeth and reinforcements therefore, jacket crowns and dental inlays and other denture work substantially as described in the examples.

15. A method of manufacturing artificial teeth and reinforcements therefor, jacket crowns, dental inlays and other denture work substantially as described in any of the examples or with reference to the drawings.

For the Applicants,
CARPMAELS & RANSFORD,
Chartered Patent Agents,
24, Southampton Buildings,
Chancery Lane,
London, W.C.2.

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This drawing is a reproduction of the Original on a reduced scale.

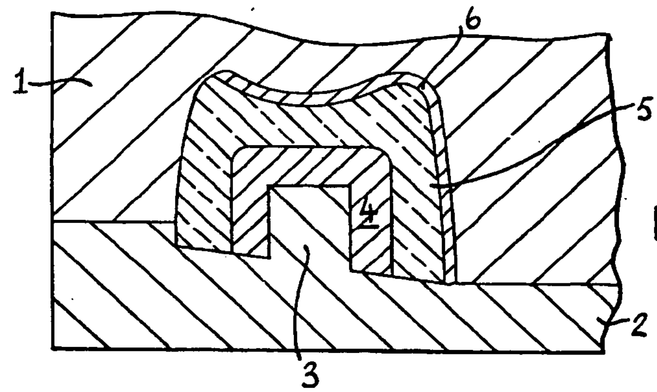

FIG. 1.

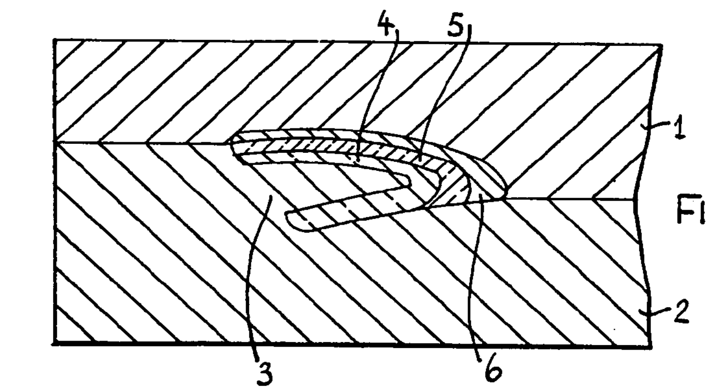

FIG. 2.

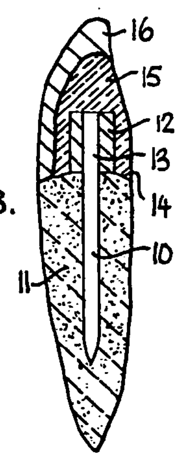

FIG. 3.

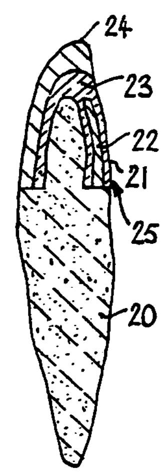

FIG. 4.

FIG. 5.